

## PATENT SPECIFICATION

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Convention Date (Germany) : June 1, 1935.

475,882

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(Patent of Addition to No. 466,042 : Dated Nov. 24, 1934.)

Complete Specification Accepted : Nov. 29, 1937.

## COMPLETE SPECIFICATION.

## Process for the Manufacture of Highly Dispersed Pigments.

We, I. G. FARBENINDUSTRIE AKTIEN-GESELLSCHAFT, a joint stock company organised under the laws of Germany, of Frankfurt-on-Main, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :—

This invention relates to the manufacture of highly dispersed pigments and is a modification of that of specification No. 32476/35. (Serial No. 466,042).

Specification No. 32476/35 (Serial No. 466,042), describes and claims compositions comprising metal containing phthalocyanines and dispersing agents, which compositions are obtained by mixing or milling metal-containing phthalocyanines with dispersing agents in the presence of water.

In accordance with the present invention metal free phthalocyanines can likewise be improved as regards softness of grain, besides clarity and intensity of shades, whereby similar advantages in practical application accrue by subjecting the same to a treatment with water soluble dispersing agents in the presence of water. This treatment can be effected at elevated temperature and is suitably carried out in a closed vessel, in which case the pressure will usually be above atmospheric. The degree of dispersion depends on the duration of the dispersing treatment, on the choice of the dispersing agents and on the temperature applied. The powdered dyestuffs obtained from these dispersions by evaporation are as stated above distinguished by their good softness of grain. The dyestuff may also be filtered with suction from the solution in order to remove a part of the dispersing agent. Prior to the dispersing treatment the metal-free phthalocyanines are preferably pasted by dissolving the same in suitable acids, such as concentrated sulphuric acid, chloresulphonic acid or trichloresulphonic acid with subsequent precipitation by means of water.

As dispersing agents such compounds are preferably employed as have at the same time a wetting capacity. Reference is made to the examples given in the

Specification of the parent patent.

The softening of the grain of the metal free phthalocyanines and their conversion into a finely divided state by means of dispersing agents can be effected merely by intensively mixing or stirring these compounds in the presence of water, if desired at an elevated temperature, or by working in a ball mill or similar device. The preparations produced by the methods described above may be applied for the colouring of artificial masses, particularly for addition to spinning solutions to be used for the production of artificial silk.

The following examples illustrate the invention, the parts being by weight :—

## EXAMPLE 1.

50 parts of the metal-free phthalocyanine obtained by heating ortho-phthalonitrile or 4-chlorophthalonitrile in the presence of formamide, are dissolved in 400 parts of concentrated sulphuric acid, poured onto ice, filtered and then rinsed. The paste thus obtained is diluted with water and boiled for 1 hour after the addition of 1 part of Turkey red oil. After separating and drying a loose, well distributable greenish-blue dyestuff powder is obtained which is especially suitable for the colouring of lacquers and for printing pastes.

## EXAMPLE 2.

50 parts of the metal-free phthalocyanine as described in the preceding example are dissolved in concentrated sulphuric acid. The solution is then poured onto ice and the precipitated dyestuff is filtered with suction. The phthalocyanine obtained is suspended in water and boiled and the paste obtained is milled in the ball or colloid mill with 2.5 parts of the sodium salt of a condensation product of formaldehyde and  $\beta$ -naphthalene sulphonie acid. The paste thus obtained is particularly suitable as wall-paper colour on account of its excellent strength.

The condensation product of formaldehyde and  $\beta$ -naphthalene sulphonie acid referred to in the preceding paragraph may be prepared by adding 4 parts of a 30% formaldehyde solution at about 60 to 100° C. to the crude mixture containing  $\beta$ -naphthalene sulphonie acid obtained

by heating 10 parts of naphthalene with 12.5 parts of a 98% sulphuric acid to about 130—140° C. The whole is then heated while stirring until free formaldehyde can no longer be traced.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. Pigment compositions comprising metal-free phthalocyanines and dispersing agents which compositions are obtained by mixing or milling metal-free phthalocyanines with water-soluble dispersing agents in the presence of water.

2. Compositions as claimed in claim 1 in the production of which the mixing or

milling is effected at an elevated temperature and/or pressure.

3. Compositions as claimed in either of the preceding claims from which a part of the liquid has been removed.

4. The compositions substantially as described in the examples.

5. The application of the preparations claimed in any of the preceding claims for the colouring of artificial masses, particularly for addition to spinning solutions to be used for the production of artificial silk.

Dated this 27th day of May, 1936.

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